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LOW EXTRACTABLE WIPERS FOR CLEANING SPACE FLIGHT HARDWARE

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ABSTRACT

There is a need for low extractable wipers for solvent cleaning of space flight hardware. Soxhlet extraction is the method utilized today by most NASA subcontractors, but there may be alternate methods to achieve the same results.

This paper will discuss the need for low NVR materials, the history of soxhlet extraction, and proposed alternate methods. The text will discuss different types of wipers, test methods, and current standards.

INTRODUCTION

Since the beginning of the space program there has been a need to clean space flight hardware with cloth wipers. In addition to using the wipers for cleaning, there was also a need to use them for collecting samples of contaminants from numerous sources in the space program. It was soon discovered that all commercial cleaning cloths (wipers) and swabs (mostly cotton) contained a wide variety and variable quantities of contaminants. These contaminants are now known as non-volatile residues (NVR's). Non-volatile residue is that quantity of molecular and particulate matter remaining, following the filtration of the solvent and evaporation of the solvent at the specified temperature. Types of NVR's include waxes, oils, grease, adhesives, and sizing compounds. When condensed, they have an oily resinous appearance.

NVR's from a wiper can be transferred to the hardware surface during a cleaning process. This is due to the fact that when exposed to a particular solvent, they are dissolved or dispersed. In this dissolved state the oils are easily transferred from the wiper to the hardware. It becomes obvious then that an item can be made no cleaner than the materials used to clean it. This applies to solvent cleanliness as well as material cleanliness. From these requirements, it may be concluded that the cleaning procedure used on the wipers is not an inconsequential matter. There are many methods of removing NVR's with great success; the most commonly used is soxhlet extraction. Soxhlet extraction utilizes a soxhlet apparatus to extract fatty or other materials with a volatile solvent. The only standard soxhlet extraction method that was found in the literature is ASTM D 1574 "Extractable Matter In Oven-Dried Wool" (ref.1). This method is, of course, not directly applicable to wipers.

The purpose of this paper is to present the methods used by different companies in extracting wipers to low NVR levels. Methods of NVR analysis are also discussed and the varying methods of gravimetric analysis are given in tabular form. It is hoped that the presentation of these various extraction and testing methods will precipitate a lively discussion that will result in the development of a standard method of testing NVR levels in wipers.

History of Soxhlet Extraction

Soxhlet extraction is named after its inventor, a German agricultural chemist, Franz von Soxhlet. A soxhlet extractor is an apparatus for use in extracting fatty or other material with a volatile solvent such as ether, alcohol, or benzene. It consists of a vertical glass cylindrical extraction tube that has both a siphon tube and a vapor tube. It is fitted at its upper end to a reflux condenser and at its lower end to a flask to allow the solvent to be distilled from the flask into the condenser. The solvent then flows back into the cylindrical tube and siphons over into the flask to be distilled again. See Figure 1.

Although many companies utilize soxhlet extraction for cleaning wipers, not all follow a standard procedure. Many of the companies use similar solvents, but the major differences are in the amount of time or number of cycles run. See Table I, "Soxhlet Extraction".

Soxhlet extraction has the advantages of requiring little attention once it is started, making use of a wide variety of solvents and continuously using hot, freshly distilled solvent for the cleaning action. There is very little solvent lost. Also, the desired NVR levels for wipers can be attained by subjecting the wipers to as many cycles as necessary. The disadvantages to using a soxhlet extractor include the long hours required for cycling, and the relatively small number of wipers that can be cleaned per batch.

The materials list and procedure used at Goddard Space Flight Center are given below. They represent the fundamentals for soxhlet extraction of wipers and swabs.

Materials and Equipment

- Polyester or cotton cleaning cloths (low linting)
- Reagent grade solvents such as ethanol, isopropanol, acetone, chloroform, trichlorotrifluoroethane
- Soxhlet extraction apparatus (available in a wide variety of sizes) which includes flask, extraction tube and water condenser
- Boiling chips or beads
- Glass fabric lined heating mantle
- Power regulator or variac for the mantle
- Rubber or plastic hose for water cooling lines
- Assorted clamps and ring stand

Procedure

1. Assemble the apparatus including the cooling water lines (make sure these are secured, wired or clamped).
2. Fill the flask about 2/3 with the solvent of choice.
3. Drop in 3 or 4 boiling aides.
4. Load the extraction tube with the swabs or cloths, not too tightly packed.
5. Fill the extraction tube about halfway to the siphon tube with the extracting solvent.
6. Assemble the apparatus and make sure the cooling water is flowing slowly.
7. Turn the power "on" and slowly increase until the boiling rate is steady and controlled.
8. Observe two or three siphoning cycles to make sure the system is working properly (make a note of the controller setting).
9. Allow the extraction to continue for 48 hours.
10. Turn off the power and allow to cool.
11. Remove the extracted material with clean, non-contaminating tongs or tweezers allowing the solvent to drain off.
12. Place in a clean pyrex or stainless steel beaker or tray.
13. Air dry in a hood or other well-ventilated, clean area.
14. Place in an oven at 100°C for about 1-2 hours, no odor of solvent should remain.
15. Store in a clean, dry, non-contaminating container or package until use.

The solvents most commonly used in extractions are methylene chloride, chloroform, isopropyl alcohol, ethanol, Freon TF, and a 3:1 mixture of 1,1,1-Trichloroethane and ethanol. Most contaminants found on assembled spacecrafts are removed by one or more of these solvents. If the cleaning cloths have been extracted by these solvents there should be a minimal chance of transference of contaminant. The wipers most commonly extracted and most commonly used in aerospace are cotton, polyester and nylon. As seen from the table, those companies which use the extracted wipers for sampling the hardware must attain lower cleanliness levels than for wipers used to clean.

A volume method of extraction has been developed by Coventry Manufacturing Company to increase the number of wipers cleaned per batch. Although the principles remain the same the process takes place on a much larger scale. Coventry Manufacturing uses a large rotating stainless steel drum to clean many wipers to acceptable cleanliness levels. The procedure is very similar to soxhlet extraction in that freshly distilled solvent is continuously being used. The difference is in the soak time. Each cycle lasts 10 minutes and is then repeated five times to achieve cleanliness levels of less than 5mg/ft². These wipers are then made available to companies which utilize them for cleaning. If wipers are needed for sampling of hardware or other materials, lower cleanliness levels can be obtained by subjecting the wipers to additional cycles.

There is another known method of volume cleaning which is used by an aerospace company. Wipers are soaked for four hours in Freon PCA with ultrasonic agitation for three minutes every half hour. With this procedure wipers which originally contained 13mg/ft² are extracted to levels of less than 3mg/ft².

Non-volatile Residue Analysis

Once the wipers have been extracted or cleaned, remaining NVR levels must be verified. There are various NVR detection techniques which are commonly used in industry. The most common method is gravimetric analysis. See Table II.

General Procedure

1. The wiper to be analyzed is soaked in a particular solvent for a specified amount of time.
2. The wiper is then removed and the solvent is evaporated to dryness in a tared weighing dish.
3. The residues remaining after evaporation are then weighed and the weight recorded per area of wiper.

Although the standard method for NVR analysis is the ASTM F-331, (ref. 2), many companies use modified versions. The fundamental differences in gravimetric NVR analysis for 15 different companies are shown in Table III. Each company has modified the procedure according to their equipment on hand and their application.

Infrared Analysis of Non-volatile Residues

Once NVR's have been attained, they can then be analyzed by IR spectroscopy. The infrared portion of the electromagnetic spectrum represents a relatively small fraction of the total known radiation span. The area of major interest within the IR is a 2.5 to 25 μ expanse. Examination of the absorption bands enables the type of contamination to be determined. The major contaminants found in wipers have been hydrocarbons, esters, and silicones. An IR spectrum is shown for each type of contaminant. See Tables IV thru VI.

CONCLUSION

This paper has discussed the general procedure and results of using a soxhlet extractor. With a soxhlet extractor aerospace specifications for wipers can be met. However, the expenses are high and the procedure time-consuming. A good alternative would be to consider commercially available wipers. However, these wipers would require monitoring of the extractable levels from batch to batch. A standardized test method for measuring extractables from wipers does not exist. Clearly, more work needs to be done in this area. Steps are being taken along these lines; the ASTM E-21 committee is in the process of standardizing a procedure for collecting NVR's deposited onto stainless steel plates in cleanrooms. Hopefully, considerations can someday be given to standardizing a test procedure for extractables from wipers.

REFERENCES

1. Standard Method of Test for Extractable Matter in Oven-Dried Wool.
ASTM Designation: D1574-66
2. Standard Test Method for Nonvolatile Residue of Halogenated Solvent Extract from Aerospace Components (Using Rotary Flash Evaporator).
ASTM Designation: F331-72.
3. Interim Federal Specification, Extraction Assembly NNN-E-00920b.
4. Contamination Control Technology. Cleaning Materials for Precision Precleaning and Use in Clean Rooms and Clean Work Stations. Military Handbook 406. p.95.
5. Product Cleanliness Levels and Contamination Control Program.
Military Standard 1246A.
6. Recommended Practices for Wipers Used in Clean Rooms and Controlled Environments. RP-4 November 1984 Draft.
7. Beeson, D. Richard: IES Lecture Notes Tutorial II. Hardware Cleaning and Sampling for Cleanliness Verification.

Table 1
Soxhlet Extraction

	1	2	3	4	5	6	7
AEROSPACE COMPANY							
NUMBER OF WIPERS CLEANED PER BATCH	50	50	6-24	16-20	25	40-50	1500
SIZE OF WIPERS	100g of material	Various sizes	Various sizes	Various sizes	9" X 9"	9" X 9"	Various sizes
SOLVENT(S) USED	Methanol-150c Chloroform or MC-150c	50% Chloroform 50% IPA	Ethanol-25c MC-25c, Ethanol-25c, MC-25c	Predistilled 3:1 Mixture of Trichloroethane and Ethanol	Freon TF or IPA	3:1 Mixture of Trichloroethane and Ethanol	Freon TMC
AMOUNT OF SOLVENT USED	2 cycles worth	2.0 liters		7.2-12.0 liters			284 liters
TYPE OF WIPERS CLEANED	Cotton	Many types		Polyester		Polyester	Cotton or polyester
NUMBER OF CYCLES	300c	N/A	100c	N/A	3-4	N/A	5
HOURS OF RUN	N/A	At least 72	96	48-80	N/A	12-18	1
CLEANLINESS LEVEL ATTAINED	0.03 mg/ft ²	4.05mg/ft ²	≤0.1 mg/ft ²	<0.5mg-0.1mg per wiper	<5mg/ft ²	<1.0mg/ft ²	<5mg/ft ²
APPLICATION	Subcontractor for aerospace companies	Subcontractor for aerospace companies	For sampling spacecraft surfaces	For sampling spacecraft hardware			Commercial wiper supplier

NOTES: Companies #1 & #7 are aerospace related companies
MC = Methylene Chloride
c = Cycles

Table II
Non-Volatile Residue Detection Techniques

TECHNIQUE	PRINCIPLE OF OPERATION	SENSITIVITY	TIME OF DETERMINATION	UNIT OF MEASUREMENT
Gravimetric	Weigh a container & evaporate a known quantity of solvent, reweigh the container, the gain is the NVR	*0.01 ppm by weight	From 0.75 to 8 hours	Milligrams per volume, converted to ppm by weight
Nephelometer	Solvent is made into an aerosol and sampled by light scattering photometer. When the NVR increases, the rate of evaporation is decreased resulting in larger aerosol droplets producing greater signal output	1 ppm	5 min.	ppm by weight or volume
Spectrophotometry	Absorption of electromagnetic radiation	1 ppm optimum	5 min. to 1 hour	percent transmittance converted to ppm or actual weight
Chromatography	Multitheoretical plate/distillation/selective adsorption	1 ppm	5 min. to 1 hour	Retention volume and direct readout are difficult

*To our knowledge these low levels cannot be attained

Table III
Gravimetric Analysis

	1	2	3	4	5	6	7
AEROSPACE COMPANY							
SIZE AND NUMBER OF WIPERS TESTED		1	1		1 wiper 3" X 3"	1 wiper 4" X 6"	1 wiper 6" X 6"
APPARATUS USED FOR SOAK	Soxhlet extractor	Beaker-Flask with Condenser	600 - ml beaker	Soxhlet extractor	Beaker	Beaker	Beaker
NUMBER OF CYCLES IN SOXHLET EXTRACTOR	10						
SOLVENT(S) USED	Chloroform or methylene chloride	Acetone, IPA or Xylene	Freon 113	3:1 mixture of trichloroethane and ethanol	Ethanol	3:1 mixture of Trichloroethane and ethanol	Trichloroethane and 10% IPA
AMOUNT OF SOLVENT	2 cycles worth	Boiling	500ml	2400ml	20-30ml		RT (25°C)
TEMPERATURE OF SOAK		10 min.	30 min.	16 hours	1 hour	2 hours	10 min.
TIME OF SOAK							Agitate every 30 min. with clean forceps
OTHER							No
FIRST EVAPORATION IS SOLVENT FILTERED?		Yes	Yes	Yes	Yes	Unknown	No
SIZE AND TYPE OF FILTER USED			.045μ				
METHOD OF EVAPORATION	Unknown	Vacuum flash evaporator	Boiling water bath	Rotary flash evaporator	Warm plate	Unknown	Oven or hot plate
TEMPERATURE OF EVAPORATION					38°C	35°-40°C	38°C
TIME OF EVAPORATION	2 hours						
SECOND EVAPORATION LAST FEW MILLILITERS EVAPORATED WITH (OR IN) HEAT LAMP			Oven	Oven			
TEMPERATURE OF EVAPORATION			105°-110°C				
TIME OF EVAPORATION	1 hour		30 min.	1-1.5 hour			

Table III
Gravimetric Analysis (Continued)

GRAVIMETRIC ANALYSIS	8	9	10	11	12	13	14	15
AEROSPACE COMPANY								
SIZE AND NUMBER OF WIPERS TESTED				2 Wipers 1 ft. 2 7" X 7"	1 Wiper 7" X 7"		100cm ²	
APPARATUS USED FOR SOAK	500ml beaker	800ml beaker	Beaker	200ml beaker	Glass tray 3" X 8" X 8"	Flask		Flash evaporator flask
NUMBER OF CYCLES IN SOXHLET EXTRACTOR								
SOLVENT(S) USED	IPA, Freon, methylene chloride or 75% trichloro 25% ethanol			3:1 mixture of trichloroethane and ethanol	Freon TF or IPA	Freon, IPA or acetone	Spectroquality 2-propanol	Various reagent grade chemicals
AMOUNT OF SOLVENT	200ml	500ml		100ml	200ml			500ml
TEMPERATURE OF SOAK					RT(25°C)	RT(25°C)	45°C	
TIME OF SOAK	1 Hour			5 Min.	10 Min.	1 Min.	30 Min.	
OTHER	Ultrasonic agitation			Ultrasonic agitation		Shake or swirl		
FIRST EVAPORATION IS SOLVENT FILTERED?	Yes	Yes	Yes	Yes	Yes	Yes		
SIZE AND TYPE OF FILTER USED	41 Whatman		0.45-5.0	0.45 Nucleopore filter	0.45 Millipore filter	Whatman		
METHOD OF EVAPORATION	Heat lamp	Steam bath	Steam bath or hot plate	Oven with a vacuum	Hot plate	Oven	Steam bath	Rotary flash evaporator
TEMPERATURE OF EVAPORATION	RT (25°)			50°C				
TIME OF EVAPORATION				2 Hours	20 Min.			
SECOND EVAPORATION LAST FEW MILLILITERS EVAPORATED WITH (OR IN)		Oven	Oven					Oven
TEMPERATURE OF EVAPORATION		110°C	110°C					105 to 110°C
TIME OF EVAPORATION	Overnight	1.5 Hour	To dryness					1.5 Hour

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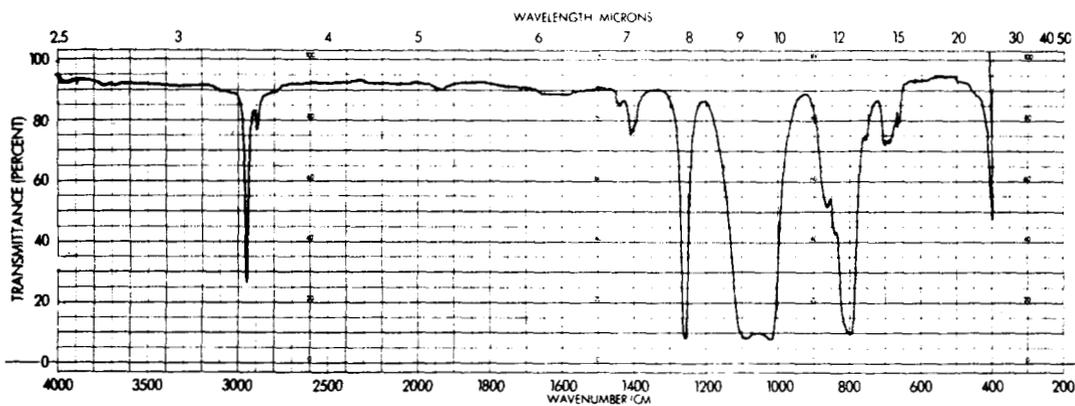


TABLE IV SILICONES

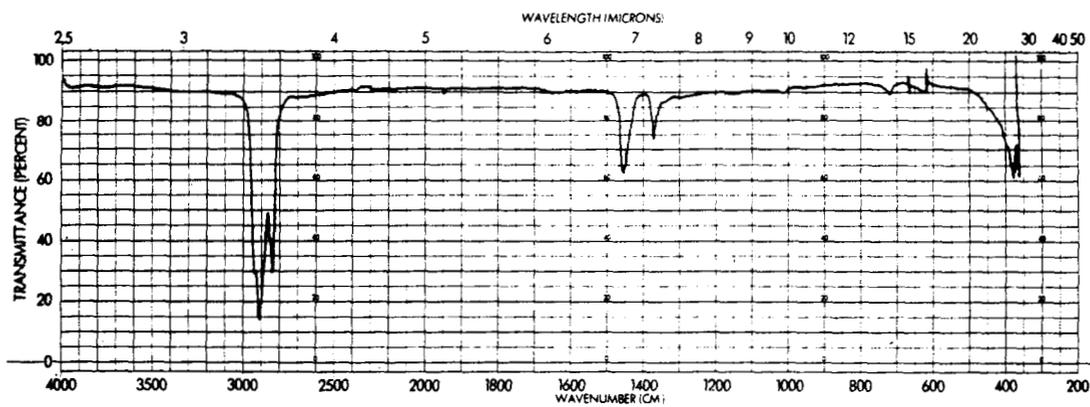


TABLE V HYDROCARBONS

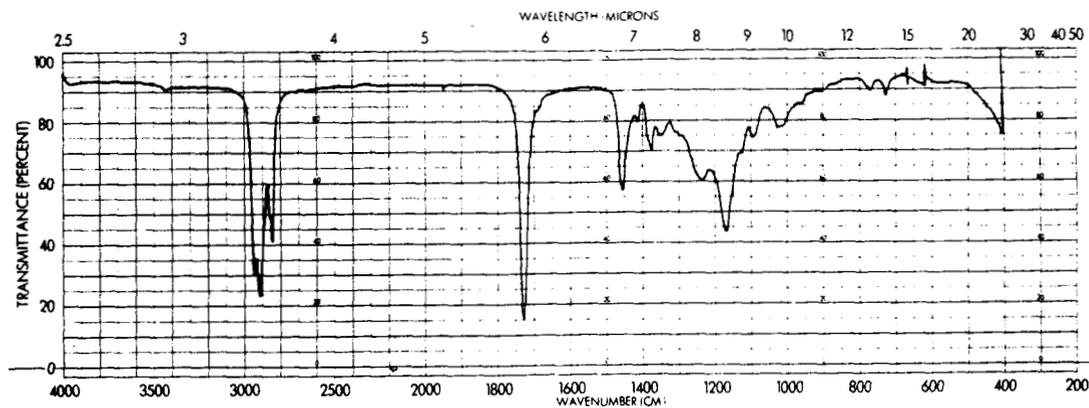


TABLE IV ESTERS

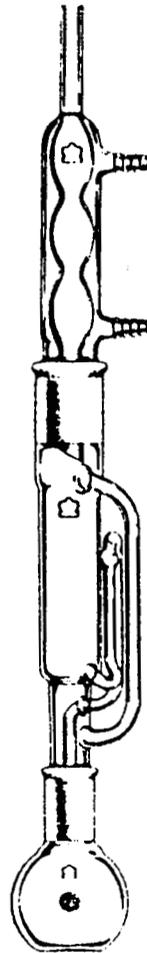


Figure 1. Soxhlet extraction apparatus